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**U.S. Army  
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Center**

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**FINAL  
WOODBIDGE RESEARCH FACILITY  
DATA VALIDATION PLAN**

SUBMITTED TO  
U.S. ARMY ENVIRONMENTAL CENTER  
ABERDEEN PROVING GROUND, MARYLAND 21010-5401

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## **Draft Woodbridge Research Facility Data Validation Plan**

### **1.0 Introduction**

The Army Environmental Center (AEC) Base Realignment and Closure (BRAC) Division tasked Horne Engineering Services, Inc. to perform data validation in support of ongoing Remedial Investigations/Feasibility Studies (RI/FS) at the Woodbridge Research Facility (WRF), Virginia. This plan presents how Horne Engineering will perform data validation for WRF site.

### **1.1 Project Objectives**

The project objective is to validate 100% of the WRF environmental analytical data to ensure that the data quality objectives described in the Quality Assurance (QA) Plan are met and that no systematic errors exist within the data sets. All field blanks, trip blanks, and equipment rinse blanks will be checked by ICF Kaiser in accordance with Section 4.4 of WRF QA Plan. The validation effort will verify that the WRF environmental data (1) are of known quality, (2) are technically valid, (3) satisfy the project objectives, and (4) are useable for their intended purpose. The data validation process will perform the following:

- Verify that no systematic errors exist within the data sets
- Assess field quality control (QC) samples to determine if sampling has adversely impacted the reported results
- Tabulate QC outliers used to assess both method and laboratory performance and determine the impact on the reported results
- Provide measures of data quality in terms of precision, accuracy, and completeness

### **1.2 Site Information**

**Woodbridge Research Facility** - The types of samples collected at WRF site include sediment/soil, water, and tissues. Sediment/soil samples were analyzed for: Metals, Pesticide/Polychlorinated biphenyls (P/PCBs), Dioxins, Volatile Organic Carbons (VOCs), Semivolatile Organic Carbons (SVOCs), and Polychlorinated Terphenyls (PCT). Water samples were analyzed for Metals, VOCs, SVOCs, P/PCBs, and PCTs. Tissues were analyzed for Metals, P/PCBs, PCTs, and PAHs.

### **1.3 Analytical Methods Used**

In accordance with AEC guidelines, EPA SW-846 and non-EPA SW-846 methods were used to characterize the site contamination. Appendix A lists the analytical methods used at the Woodbridge Research Facility RI/FS.

### **2.0 Data Validation Methodology**

Horne Engineering will validate 100% of the environmental analytical data to ensure that the data quality objectives are met using a combination of electronic and manual methods. In addition, Horne Engineering will validate the analytical data used for risk assessment fully manually, an effort equivalent to an EPA CLP validation. Figure 1 illustrates Horne Engineering data validation flowchart.

#### **2.1 Electronic and Manual Validation for 100% of the Data**

##### **2.1.1 Data Validation Requirements**

Data will be validated to meet the requirements specified in the August 9, 1996 Commonwealth of Virginia, Department of Environmental Quality memo on the WRF RI/FS Revised Data Validation Plan. Specifically, the QC elements (1) matrix spike and duplicate (MS/MSD) recoveries, (2) surrogate spike recoveries, and (3) detection level will be used to validate the analytical data. This level of validation is based on the reported laboratory results only.

##### **2.1.2 Data Submittal Requirements**

The laboratory will be requested to provide these QC information electronically and/or hardcopy in a prescribed format. For the data that can not be provided electronically, the laboratory will be requested to submit the hardcopy of the data in a prescribed format on summary data forms<sup>1</sup>.

It is the laboratory's responsibility to ensure that the information contained in the summary data forms and electronic data deliverable is accurate and complete. Horne Engineering will check the accuracy of the electronic and hardcopy deliverables by comparing the information in the summary forms (hard copy) to that presented electronically (such as sample name, sample results, blank contaminants, and etc.). The data will be considered usable if the information in the electronic deliverable and summary forms (hard copy) are consistent. If the information varies, Horne Engineering will work with the laboratory and prime contractor to correct the deficiencies. Horne Engineering will notify AEC of any potential impact this may have on the

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<sup>1</sup> Summary Forms: Forms that summarize QC results (based on method and/or EPA Region III data validation requirements). Examples of summary forms can be found in OLM01.8 (*Organic*) and ILM04.0 (*Inorganic*) CLP SOW.

project schedule. Horne Engineering will not proceed with data validation activities until the data deliverables are deemed to be acceptable.

Horne Engineering will perform this check on the first two lots and periodically throughout the project. Horne Engineering will perform these checks at a maximum for a total of 6 lots.

### **2.1.3 Electronic and Manual Validation**

Horne Engineering will perform electronic and manual data validation for the following criteria - matrix spike and duplicate (MS/MSD) recoveries, surrogate spike recoveries, and detection level in accordance with Department of Environmental Quality requirements. The qualifiers will be assigned to the analytical data if the samples data does not meet the QC requirements.

Before use, the electronic data validation system will be verified by comparing the results of manual verification of the first two lots. If the validation results do not match, the electronic data validation system will be modified.

Horne Engineering will also test the accuracy of data deliverables by performing fully manual data validation on the first two lots (for each fraction). The data reported in the summary and batch reports should be the same and consistent with the raw data. In case of any discrepancies, AEC will be notified for further instructions.

### **2.1.4 Data Validation Summary**

A data validation Summary will be provided after reviewing the data. The data validation summary will briefly describe the overall data quality. The summary will identify the reviewed data quality.

The data validation Summary will include the following:

- A narrative including a statement that briefly summarizes the overall data quality
- Data summary table
- IRDMIS data entry on each sample result
- Supporting Documentation including all documentation needed to substantiate the findings described in the narrative.

## **2.2 Fully Manual Data Validation for Analytical Data used in Risk Assessment**

Horne Engineering will perform a fully manual data validation for the analytical data used in the risk assessment in accordance with EPA Region III requirements. Data will be qualified manually by reviewing additional QC elements such as raw data, chromatograms, and calculation verification. Table 1 lists the samples to be validated fully manually.

Table 1: Number of Analytical Samples to be Validated Fully Manually

Analyte/Media	Surface Water	Sediment	Surface Soil	Ground Water
VOCs	13	16	19	17
SVOCs	16	17	20	16
P/PCBs	17	50	81	77
PCTs	10	17	8	9
PAHs	16	41	61	57
Metals	58	52	60	59
Total # of Samples	130	193	249	235

\* Soil samples include tissue samples.

### 2.2.1 Data Validation Requirements

Appendix B specifies the data elements required to perform data validation in accordance with QA Plan. The laboratory will be requested to provide this information in a prescribed format and raw data.

The QC requirements for each method are summarized in the tables and are included in Appendix C. These tables present the validation qualifiers for each method. In general, the following QC requirements will be reviewed.

- Sample holding Times
- Instrument Performance Check
- Initial Calibration
- Continuing Calibration
- ICP Serial Dilutions
- Blanks
- Matrix Spikes and Duplicate (MS/MSD) Recoveries
- Surrogate Spike Recoveries
- Internal Standard Areas and Retention Times

Horne Engineering assumes that the ESE laboratory is going to provide these information either electronically or in summary forms (hard copy).

## **2.2.2 Data Submittal Requirements**

To perform fully manual data validation, Horne Engineering will request a complete hard copy of the data package. The following lists the major items that are required to be included in the hard copy data packages:

- Traffic report/chain of custody
- Summary forms
- Documentation of sample preservation and transport
- Documentation that the analytical results are in control and within the certified range
- Documentation on traceability of instrument standards, samples, and data
- Documentation on sample analysis (e.g., providing raw data, mass spectrometer chromatograms, reconstructed ion chromatogram)
- Documentation that verifies sample calculation and that no systematic errors exist within the data sets
- Documentation listed in section 2.2.1

## **2.2.3 Data Validation Summary**

A Data Validation Summary will be prepared. This summary will include the following:

- A narrative including a statement that briefly summarizes the overall data quality
- Data summary table: Includes all results with appropriate qualifiers if applicable..
- IRDMIS data entry on each sample result
- Supporting Documentation including all documentation needed to substantiate the findings described in the narrative..

## **2.3 Data Validation Quality Assurance and Quality Control**

All validated data will be subject to QA/QC. The purpose is to ensure that the data validation project objectives are met. Appendix D describes the standard operating procedures (SOPs) developed by Horne Engineering to meet data validation QA/QC objectives.

## **3.0 Quality Assurance Report**

For each site, a QA Report will be prepared that includes the following:

- Narrative
  - A statement that defines the level of the data review, overview, and data quality summary (per site) using data quality reports for each lot
  - Major and minor problems associated with the samples in each site
- Attachments

- Data summary table (per lot): Includes all results with appropriate qualifiers if applicable
- IRDMIS data entry on each sample
- Supporting documentation (per lot) including all documentation needed to substantiate the findings described in the narrative

Appendix E contains an example of a QA report.

#### **4.0 Schedule**

A draft schedule is provided in Appendix F. This schedule is dynamic and could be routinely modified to reflect the project activities and progresses.



## 5.0 Reference Documents

EcoChem, Inc. 1990. *Jefferson Proving Ground South of the Firing Line, Technical Memory Data Quality Assessment Procedures*, June 12, 1995.

EPA 1993 *Contract Laboratory Program National Functional Guidelines for Organic Data Review*.

EPA 1993 *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*.

EPA 1991 *Contract Laboratory Program Statement of Work for Organic Analysis*.

EPA 1991 *National Functional Guidelines for Organic Data Review* (Draft December 1990, as revised June 1991).

AEC 1996 *Sampling and Analysis Plan, Quality Assurance Project Plan (Volume II) for Woodbridge Research Facility RI/FS*.

EPA 1994 *Region III Modifications to the National Functional Guidelines for Organic Data Review*.

EPA 1993 *Region III Modifications to the National Functional Guidelines for Evaluation Inorganic Data Review*.

EPA 1990 *Guidance for Data Usability in Risk Assessment Interim Final*.

# **Appendix A**

## **Analytical Method Used at the Woodbridge Research Facility RI/FS**

### Summary of Analytical Methods

Parameter/Analytes		Matrix	Reference USEPA Method
TCL Volatile Organic Compounds		Aqueous/Soil	USEPA SW-846 M8240
TCL Semivolatile Organic Compounds		Aqueous/Soil	USEPA SW-846 M8270
Polynuclear Aromatic Hydrocarbons		Aqueous/Soil	USEPA SW-846 M8310
TCL Pesticides/PCBs		Aqueous/Soil	USEPA SW-846 M8080
Polychlorinated Terphenyls		Aqueous/Soil	Hale, et. al.
Metals	TAL Metals	Soil	USEPA SW-846 M6010
		Aqueous	USEPA SW-846 M6010
	Arsenic	Soil	USEPA SW-846 M6010
		Aqueous	USEPA SW-846 M6020
	Antimony	Soil	USEPA SW-846 M7041
		Aqueous	USEPA SW-846 M6020
	Beryllium	Soil	USEPA SW-846 M6020
		Aqueous	USEPA SW-846 M6020
	Cadmium	Soil	USEPA SW-846 M6010
		Aqueous	USEPA SW-846 M6020
	Lead	Soil	USEPA SW-846 M6010
		Aqueous	USEPA SW-846 M6020
	Mercury	Soil	USEPA SW-846 M7471
		Aqueous	USEPA SW-846 M7470
	Thallium	Soil	USEPA SW-846 M6010
		Aqueous	USEPA SW-846 M6020

# **Appendix B**

## **Data Submittal Requirements**

## Method 8240 -- VOA

The following information is needed for each bulletin:

### ▶ **Hardcopy of Traffic Report / Chain of Custody**

- Sampling Date;
- Laboratory Received Date /Signature;
- Lot #, Matrix;
- List of all the Samples included in the Lot;
- Determination of Trip/Equipment/Rinsate blanks, duplicate, and MS/MSD samples.

### ▶ **Sample Result Summary**

- Lab name, lot #, matrix, sample wt/vol (depending on the matrix), %moisture (for soil samples), date received and analyzed, time analyzed, and dilution factor;
- List of the target compounds;
- Found/Calculated Concentration/ Concentration Units;
- "U" flag for non-detected compounds.

*Note: The above information should be provided for samples, matrix spike (MS), matrix spike duplicate (MSD), and blanks.*

### ▶ **System Monitoring Compounds Recovery**

- % Recovery of toluene-d8, bromofluorobenzene, and 1,2-Dichloroethane-d4 for all samples, QC samples (MS/MSD), blanks, and duplicates.

### ▶ **MS/MSD Recoveries**

- Spike added, sample concentration (ug/L or ug/kg depending on the matrix), MS and MSD concentrations, MS and MSD % recoveries, and RPD for the spiked compounds (1,1-Dichloroethene, trichloroethane, benzene, toluene, and chlorobenzene).

### ▶ **Instrument Performance Check (BFB)**

- Lab File ID, Instrument ID, BFB Injection Time and Date;
- List of all the calibration (initial and/or continuing), blank(s), and samples analyzed under this tune along with their date and time of analysis;
- List of all the required m/e, ion abundance criteria, and % relative abundance for the tune. (Refer to the USEPA CLP OLM01.8 for the tune requirements).

► **Method Blank Summary**

- Instrument ID and Matrix;
- Method blank date analyzed;
- Method blank time analyzed;
- List of all the samples and QC samples (MS/MSD) associated with the method blank.

► **Initial Calibration Results**

- Instrument ID, Start and End Date of initial calibration;
- Lab File ID for each calibration standard;
- RRF (for each calibration standard), average RRF, and %RSD for all target and surrogate compounds.

► **Continuing Calibration Results**

- Instrument ID, Date and Time of continuing calibration, Start and End of associated Initial Calibration;
- Average RRF (from the associated initial calibration), RRF50, and %D for target and surrogate compounds.

► **Internal Standard Area and RT Summary for each Continuing Calibration**

- Lab File ID (continuing calibration) and Instrument ID;
- Date and Time of continuing calibration analysis;
- Internal Standard Area and RT for each continuing calibration;
- Internal Standard Area and RT for all the associated samples, blanks, and QC samples (MS/MSD).

## Method 8270 -- SVOA

The following information is needed for each bulletin:

### ► **Hardcopy of Traffic Report / Chain of Custody**

- Sampling Date;
- Laboratory Received Date /Signature;
- Lot #, Matrix;
- List of all the Samples included in the Lot;
- Determination of Equipment/Rinsate blanks, duplicate, and MS/MSD samples.

### ► **Sample Result Summary**

- Lab name, lot #, matrix, sample wt/vol (depending on the matrix), %moisture (for soil samples), date received, extracted, and analyzed, time analyzed, and dilution factor;
- List of the target compounds;
- Injection Volume (ul) and Concentrated Extract Volume;
- Found/Calculated Concentration/ Concentration Units;
- "U" flag for non-detected compounds.

*Note: The above information should be provided for samples, matrix spike (MS), matrix spike duplicate (MSD), and blanks.*

### ► **Surrogates Recovery**

- % Recovery of nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14, phenol-d5, 2-fluoropropenol, 2,4,6-tribromophenol, 2-chlorophenol-d14, and 1,2-dichlorobenzene-d4 for all samples, QC samples (MS/MSD), blanks, and duplicates.

### ► **MS/MSD Recoveries**

- Spike added, sample concentration (ug/L or ug/kg depending on the matrix), MS and MSD concentrations, MS and MSD % recoveries, and RPD for the spiked compounds (phenol, 2-chlorophenol, 1,4-dichlorobenzene, n-nitroso-di-n-propylamine, 1,2,4-trichlorobenzene, 4-chloro3-methylphenol, acenaphthene, 4-nitrophenol, 2,4-dinitrotoluene, pentachlorophenol, and pyrene)

### ► **Instrument Performance Check (DFTPP)**

- Lab File ID, Instrument ID, DFTPP Injection Time and Date;
- List of all the calibration (initial and/or continuing), blank(s), and samples analyzed under this tune along with their date and time of analysis;
- List of all the required m/e, ion abundance criteria, and % relative abundance for the tune. (Refer to the USEPA CLP OLM01.8 for the tune requirements).

► **Method Blank Summary**

- Instrument ID and Matrix;
- Method blank date extracted and analyzed;
- Method blank time analyzed;
- List of all the samples and QC samples (MS/MSD) associated with the method blank.

► **Initial Calibration Results**

- Instrument ID, Start and End Date of initial calibration;
- Lab File ID for each calibration standard;
- RRF (for each calibration standard), average RRF, and %RSD for all the required target and surrogate compounds.

► **Continuing Calibration Results**

- Instrument ID, Date and Time of continuing calibration, Start and End of associated Initial Calibration;
- Average RRF (from the associated initial calibration), RRF50, and %D for target and surrogate compounds.

► **Internal Standard Area and RT Summary for each Continuing Calibration**

- Lab File ID (continuing calibration) and Instrument ID;
- Date and Time of continuing calibration analysis;
- Internal Standard Area and RT for each continuing calibration;
- Internal Standard Area and RT for all the associated samples, blanks, and QC samples (MS/MSD).



## Method 8080/8081 --- Pesticide/PCB

The following information is needed for each bulletin:

### ▶ **Hardcopy of Traffic Report/ Chain of Custody**

- Sampling Date;
- Laboratory Received Date/Signature;
- Lot #, Matrix;
- List of all the Samples included in the Lot;
- Determination of Equipment/Rinsate blanks, MS/MSD samples, and duplicates.

### ▶ **Sample Result Summary**

- Lab name, lot #, matrix, sample wt/vol (depending on the matrix), %moisture (for soil samples), date received , extracted, and analyzed, time analyzed, and dilution factor;
- List of the target compounds;
- Column ID, Extraction Procedure;
- Injection Volume (ul) and Concentrated Extract Volume;
- Found/Calculated Concentration/ Concentration Units;
- Cleanup (Florisil, GPC, Sulfur, or Silica Gel Cleanup);
- "U" flag for non-detected compounds.

*Note: The above information should be provided for samples, matrix spike (MS), matrix spike duplicate (MSD), and blanks (method and instrument blanks), duplicates (if applicable) on each column.*

### ▶ **Surrogates Recovery**

- % Recovery of TCX and DCB for all samples, QC samples (MS/MSD), blanks, and duplicates. % Recoveries should be provided for each column separately.

### ▶ **MS/MSD Recoveries**

- Spike added, sample concentration (ug/L or ug/kg depending on the matrix), MS and MSD concentrations, MS and MSD % recoveries, and RPD for spiked compounds ( Lindane, Heptachlor, Aldrin, Dieldrin, Endrin, and 4,4'-DDT).

### ▶ **Method Blank Summary**

- Instrument ID (1)/(2), Column ID (1)/ (2), and Matrix;
- Method blank date extracted and analyzed;
- Method blank time analyzed;
- List of all the samples and QC samples (MS/MSD) associated with the method blank.

► **GC/ECD Instrument Performance Check**

- %Resolution of the analytes in the resolution check mixture for column (1)/(2);
- Analysis of performance evaluation mixture (PEM) immediately after the resolution check mixture;
- Relative Percent Difference (RPD) between the calculated amount and the true amount for each of compounds and surrogates in the PEM;
- RT of compounds in the PEM for column (1)/(2);
- 4,4'-DDT % breakdown, Endrin % breakdown, combined % breakdown in PEM for column (1)/(2);

► **Initial Calibration Results**

- Instrument ID(1)/(2), Date(s) Analyzed,
- RT for each initial calibration, mean RT , RT window {column (1)/(2)} for all target compounds and surrogates;
- Linear correlation coefficient for column (1)/(2);
- Initial calibration sequence.

► **Continuing Calibration Results**

- Instrument ID (1)/(2), Date and Time of continuing calibration on column (1)/(2);
- RT and RT window for each compound in continuing calibration {column (1)/(2)};
- Response factor for continuing calibration for column (1)/(2);
- Lab sample ID for the associated instrument blank (PIBLK) for column (1)/(2);
- Date and time of associated instrument blank analysis for column (1)/(2);
- Associated initial calibration date for column (1)/(2);
- Analysis of a performance evaluation mixture (PEM), or a continuing calibration standard (CCS= IND6) and an instrument blank at least every 12 hours and at the end of the analytical run for column (1)/(2).

*Note: The above information should be provided for each continuing calibration (PEM and IND6).*

► **Analytical Sequence**

- List of resolution check mixture, calibrations, samples, and blanks in chronological order (report date and time of analysis ) for each run for column (1)/(2);
- Surrogate RT for all samples, blanks, and MS/MSD samples for column (1)/(2);

► **Pesticide Cleanup Check (Florisil/GPC/Silica-gel/Sulfur Cleanup)**

- %Recovery of the compounds (depending on what kind of cleanup procedure has been done).

## Method 6010/6020/7041/7471/7470 --- Inorganic

The following information is needed for each bulletin:

### ▶ **Hardcopy of Traffic Report/ Chain of Custody**

- Sampling Date;
- Laboratory Received Date/Signature;
- Lot #, Matrix;
- List of all the Samples included in the Lot;
- Determination of Trip/Equipment/Rinsate blanks, spikes, and duplicates.

### ▶ **Sample Result Summary**

- Lab name, lot #, matrix, %moisture (for soil samples), date received and analyzed, time analyzed, pH, and dilution factor;
- List of the analytes;
- Concentration, concentration units (ug/L or mg/kg);
- Method type;
- "U" flag for non-detected compounds;
- Initial sample weight or volume and final volume.

*Note: The above information should be provided for samples, spikes, and duplicates.*

### ▶ **Initial and Continuing Calibration Verification (ICV/CCV)**

- Initial and continuing calibration source;
- True value, found value, and %R for initial calibration and each continuing calibration verification;
- Linear correlation coefficient for initial calibration;

### ▶ **ICP Interference Check Solution (ICP-A/ ICP-AB)**

- True value of Sol. A and Sol AB;
- Initial and found values of Sol A and Sol AB ;
- ICP Instrument ID.

### ▶ **Blanks (Preparation Blank (PB)) / Initial Calibration Blank (ICB) / Continuing Calibration Blank(CCB)**

- PB matrix and units;
- Concentration of target elements for PB; ICB;
- Concentration of target elements for all the CCBs in the run.

► **Spike Sample Recovery**

- Spike Sample Result (SSR), Sample Result (SR), and Spike Added (SA) for each analyte;
- %Recovery ;
- Matrix, %solids (if matrix is soil).

► **Post Digest Spike Sample Recovery**

- Spike Sample Results (SSR), Sample Result (SR), and Spike Added (SA) for analytes that required post digestion;
- %Recovery.

► **Duplicates**

- %Solids for sample and duplicate (if matrix is soil);
- Sample and duplicate concentrations, concentration units (ug/L or mg/kg);
- RPD , Control Limit, and Method used.

► **Analytical Sequence**

- Provide analytical sequence of standards, ICV, ICB, ICSA (initial and final), ICSAB( initial and final),CCVs, CCBs, PB, LCS, Spike, Duplicate, Samples, and Serial Dilutions according to the method for each run (batch);
- Dilution Factor for each sample in the run.
- Date and Time of each analysis.

► **Laboratory Control Sample (LCS)**

- Solid LCS Source, Liquid LCS Source;
- True value, Found value, %R , limits (for soil only);
- Concentration units (ug/l for aqueous and mg/kg for soil).

► **ICP Serial Dilution (ICP only)**

- Initial Sample Result (I), Serial Dilution Result (S), and %Difference;
- Matrix and concentration units.

▶ **Instrument Detection Limits**

- Flame AA ID Number, Furnace AA ID Number, and ICP ID Number;
- Instrument Detection Limit (IDL) in ug/L;
- Reporting Limit (ug/L);
- Analysis Method.

▶ **Preparation Log**

- Analysis Method;
- Preparation Date, Weight (gram), Volume (ml).

▶ **Standard Addition Result (Furnace only)**

- Sample Number
- Concentration in ug/L for zero, first, second, and third addition;
- Absorbance for each addition;
- Final concentration;
- Linear correlation coefficient.

## Method 8310 --- PAH

The following information is needed for each bulletin:

### ► **Hardcopy of Traffic Report / Chain of Custody**

- Sampling Date;
- Laboratory Received Date /Signature;
- Lot #, Matrix;
- List of all the Samples included in the Lot;
- Determination of Equipment/Rinsate blanks, duplicate, and MS/MSD samples.

### ► **Sample Result Summary**

- Lab name, lot #, matrix, sample wt/vol (depending on the matrix), %moisture (for soil samples), date received , extracted, and analyzed, time analyzed, and dilution factor;
- List of the target compounds;
- Injection Volume (ul) and Concentrated Extract Volume;
- Reported Concentration/ Concentration Units;
- "U" flag for non-detected compounds.

*Note: The above information should be provided for samples, matrix spike (MS), matrix spike duplicate (MSD), blanks (method and instrument blanks), and field duplicates (if any analyzed).*

### ► **Surrogates Recovery**

- % Recovery of triphenylene for all samples, QC samples (MS/MSD), blanks, and duplicates.

### ► **MS/MSD Recoveries**

- Spike added, sample concentration (ug/L or ug/kg depending on the matrix), MS and MSD concentrations, MS and MSD % recoveries, and RPD for the spiked compounds (acenaphthene, acenaphthylene, anthracene, benzo(a)pyrene, benzo(k)pyrene, fluorene, naphthalene, and phenanthrene)

### ► **Method Blank Summary**

- Instrument ID and Matrix;
- Method blank date extracted and analyzed;
- Method blank time analyzed;
- List of all the samples and QC samples (MS/MSD) associated with the method blank.

► **Initial Calibration Results**

- Instrument ID, Start and End Date of initial calibration;
- RT, mean RT , and RT windows for each analyte and surrogate;
- Linear correlation coefficient;
- Initial calibration sequence;

► **Continuing Calibration Results**

- Instrument ID, Date and Time of continuing calibration;
- RT and RT window for each analyte and surrogate in continuing calibration;
- Response factor for continuing calibration;
- *Analysis of a continuing calibration and an instrument blank at least once every 12 hours within the analytical run and once at the end of the run.*

► **Analytical Sequence**

- List of analytical sequence in chronological order according to the method;
- RT of surrogate in samples, blanks, MS, and MSD.

## POLYCHLORINATED TERPHENYLS (PCT)

The following information is needed for each bulletin:

### ► **Hardcopy of Traffic Report/ Chain of Custody**

- Sampling Date;
- Laboratory Received Date/Signature;
- Lot #, Matrix;
- List of all the Samples included in the Lot;
- Determination of Equipment/Rinsate blanks, MS/MSD samples, and duplicates.

### ► **Sample Result Summary**

- Lab name, lot #, matrix, sample wt/vol (depending on the matrix), %moisture (for soil samples), date received, extracted, and analyzed, time analyzed, and dilution factor;
- List of the target compounds;
- Column ID, Extraction Procedure;
- Injection Volume (ul) and Concentrated Extract Volume;
- Concentration/ Concentration Units;
- Cleanup (Acid / Silica Gel Cleanup);
- "U" flag for non-detected compounds.

*Note: The above information should be provided for samples, matrix spike (MS), matrix spike duplicate (MSD), and blanks (method and instrument blanks), duplicates (if applicable) on each column.*

### ► **Surrogates Recovery**

- % Recovery of TCX and DCB for all samples, QC samples (MS/MSD), blanks, and duplicates. % Recoveries should be provided for each column separately.

### ► **MS/MSD Recoveries**

- Spike added, sample concentration (ug/L or ug/kg depending on the matrix), MS and MSD concentrations, MS and MSD % recoveries, and RPD for spiked compounds (Aroclor 5432 and Aroclor 5460).

### ► **Method Blank Summary**

- Instrument ID (1)/(2), Column ID (1)/ (2), and Matrix;
- Method blank date extracted and analyzed;
- Method blank time analyzed;
- List of all the samples and QC samples (MS/MSD) associated with the method blank.



► **GC/ECD Instrument Performance Check**

- %Resolution of the analytes in the resolution check mixture for column (1)/(2);
- Analysis of performance evaluation mixture (PEM) immediately after the resolution check mixture;
- Relative Percent Difference (RPD) between the calculated amount and the true amount for each of compounds and surrogates in the PEM;
- RT of compounds in the PEM for column (1)/(2);
- 4,4'-DDT % breakdown, Endrin % breakdown, combined % breakdown in PEM for column (1)/(2);

► **Initial Calibration Results**

- Instrument ID(1)/(2), Date(s) Analyzed,
- RT for each initial calibration, mean RT , RT window {column (1)/(2)} for target compounds and surrogates;
- Linear correlation coefficient for column (1)/(2);

► **Daily Calibration Standard**

- Analysis of daily calibration standard every 12 hours;
- Linear correlation coefficient for column (1)/(2).

► **Continuing Calibration Results**

- Instrument ID (1)/(2), Date and Time of continuing calibration on column (1)/(2);
- RT and RT window for each compound in continuing calibration {column (1)/(2)};
- Response factor for continuing calibration for column (1)/(2);
- Lab sample ID for the associated instrument blank (PIBLK) for column (1)/(2);
- Date and time of associated instrument blank analysis for column (1)/(2);
- Associated initial calibration date for column (1)/(2);
- Analysis of a continuing calibration standard (CCS) and an instrument blank at least every 12 hours.

► **Analytical Sequence**

- List of resolution check mixture, calibrations, samples, and blanks in chronological order (report date and time of analysis ) for each run for column (1)/(2);
- Surrogate RT for all samples, blanks, and MS/MSD samples for column (1)/(2);

► **Pesticide Cleanup Check (Florisil/GPC/Silica-gel/Sulfur Cleanup)**

- %Recovery of the compounds (depending on what kind of cleanup procedure has been done.

# **Appendix C**

## **Quality Assurance Requirements, Criteria and Validation Qualifiers**

**TABLE 1**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(VOA QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	Validation Qualifier															
Initial Calibration 6-pt curve	Set-up, major maintenance, and quarterly	RRF ≥ 0.01 and RSD ≤ 30	If RRF<0.05, then Detected Results--- L and Nondetected Results --- R If RSD>30% then, Detected Results ---J, Nondetected Results will be estimated UJ if RSD>50%.															
Holding Time	NA	Water/Soil: 14 days to analysis (if preserved)	Detected Results----L, Nondetected---UL If holding time is grossly exceeded: Detected---L, Nondetected---R															
Daily Calibration standard/ Continuing Calibration standard	Every 12 hours	%D ≤ 25, RRF ≥ 0.05	If RRF<0.05, then Detected Results--L Nondetected Results---R If %D>25%, then Detected Results--J, Nondetected---UJ if %D>50.															
Method Blank	Every 12 hours and after continuing calibration	No target analytes	Use Region III Modifications to NFGs for Organic Data Review to qualify the detected results ---B.															
Tuning	Prior to calibration	Must meet tuning criteria in USEPA CLP OLM01.8.	Professional Judgement															
Internal Standard(IS)	Every Sample	RT = ±30 seconds, Area changes by a factor of two (-50% to +100%)	If <u>IS area is out</u> , then Detected results ---J, Nondetected----UJ. If extremely low area counts are reported, then Nondetected---R. If IS RT is out, use <u>professional Judgement</u> .															
System Monitoring Compound	Every sample, including QC samples	<table><tr><td></td><td>Water</td><td>Soil</td></tr><tr><td></td><td>%Recoveries</td><td>%Recoveries</td></tr><tr><td>Toluene-d8</td><td>88-110</td><td>84-138</td></tr><tr><td>Bromofluorobenzene</td><td>86-115</td><td>59-113</td></tr><tr><td>1,2-Dichloroethane- d4</td><td>76-114</td><td>70-121</td></tr></table>		Water	Soil		%Recoveries	%Recoveries	Toluene-d8	88-110	84-138	Bromofluorobenzene	86-115	59-113	1,2-Dichloroethane- d4	76-114	70-121	Use Region III Modifications to NFGs for Organic Data Review to qualify samples if %Recovery is out.
	Water	Soil																
	%Recoveries	%Recoveries																
Toluene-d8	88-110	84-138																
Bromofluorobenzene	86-115	59-113																
1,2-Dichloroethane- d4	76-114	70-121																

**TABLE 1**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(VOA QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria					Validation Qualifier	
Matrix Spike and Duplicates	1 per lot/ matrix/level	Spikes 1,1-dichloroethene Trichloroethene Benzene Toluene Chlorobenzene	%R(Water) 61-145 71-120 76-127 76-125 75-130	%R(Soil) 59-172 62-137 66-142 59-139 60-133	RPD(Water) ≤14 ≤14 ≤11 ≤13 ≤13	RPD(Soil) ≤22 ≤24 ≤21 ≤21 ≤21	Use professional judgement to determine the need for some qualification of the data using the MS/MSD results in conjunction with other QC criteria.	

\* 1,1-dichloroethene  
\*\* trichloroethane

**TABLE 2**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(SVOA QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	Validation Qualifier
Initial Calibration curve	Set-up, major maintenance	RRF $\geq$ 0.05 and RSD $\leq$ 30%	If RRF < 0.05 and Results Detected then ---J and Nondetected Results ---R If RSD > 30% then, Detected Results ---J, Nondetected Results will be estimated UJ if RSD > 50%.
Holding Time	NA	Water/Soil: 7 days to extraction and 40 days after extraction to analysis date.	Detected Results ---J or L, Nondetected ---UJ or UL (L and UL qualifiers will be used if loss of Semivolatile compounds is evident due to exceeding the holding time criteria). If holding time is grossly exceeded: Detected ---J, Nondetected ---R or UJ
Daily Calibration standard/ Continuing Calibration standard	Every 12 hours	%D $\leq$ 25, RRF $\geq$ 0.05	If RRF < 0.05, then Detected Results ---J Nondetected Results ---R If %D > 25%, then Detected Results ---J, Nondetected ---UJ if %D > 50.
Method Blank	Every 12 hours and after continuing calibration	No target analytes	Use Region III Modifications to NFGs for Organic Data Review to qualify the detected results ---B.
Tuning	Prior to calibration	Must meet tuning criteria in USEPA CLP OLM01.8.	Professional Judgement
Internal Standard(IS)	Every Sample	RT = $\pm$ 30 seconds, Area changes by a factor of two (-50% to +100%)	If IS area is out, then Detected results ---J, Nondetected ---UJ. If extremely low area counts are reported, then Nondetected ---R. If IS RT is out, use professional judgement

**TABLE 2**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(SVOA QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	%R - Water	%R - Soil	Validation Qualifier
Surrogate spikes	Every sample, including QC samples	Surrogates nitrobenzene-d5 2-Fluorobiphenyl Terphenyl-d14 Phenol-d5 2-Fluoroprophenol-- 2,4,6Tribromophenol 2-Chlorophenol-d4 1,2Dichlorobenzene-d4	35-144 43-116 33-141 10-110 21-110 10-123 33-110* 16-110* * advisory	23-120 30-115 18-137 24-113 25-121 19-122 20-130* 20-130* * advisory	If any two base/neutral or acid surrogates are out of specification, or if any one base/neutral or acid extractable surrogate has a recovery of less than 10%, then qualify samples using Region III Modifications to NFGs for Organic Data Review.
Matrix Spike and Duplicates	1 per lot/matrix/level	Compound phenol 2-chlorophenol 1,4-dichlorobenzene n-nitroso-di-n-propylamine 1,2,4-Trichlorobenzene 4-chloro-3-methylphenol acenaphthene 4-nitrophenol 2,4-dinitrotoluene pentachlorophenol pyrene	%R/RPD-Water 12-110 / ≤42 27-123 / ≤40 36-97 / ≤28 41-116 / ≤38 39-98 / ≤28 23-97 / ≤42 46-118 / ≤31 10-80 / ≤50 24-96 / ≤38 9-103 / ≤50 26-127 / ≤31	%R/RPD-Soil 26-90 / ≤35 25-102 / ≤50 28-104 / ≤27 41-126 / ≤38 38-107 / ≤23 26-103 / ≤33 31-137 / ≤19 11-114 / ≤50 28-89 / ≤47 17-109 / ≤47 35-142 / ≤36	Use professional judgement to determine the need for some qualification of the data using the MS/MSD results in conjunction with other QC criteria.

**TABLE 3**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Pesticide/PCB QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance QC	Validation Qualifier
Initial calibration curve single component, multi-component	Set-up, major maintenance	$r \geq 0.995$ r: linear correlation coefficient	Detected results ---J Professional Judgement on Nondetected results
Daily calibration standard	12 hours	$r \geq 0.995$	Detected results ---J Professional Judgement on Nondetected results
Independent reference standard (calibration check)	Weekly	Recovery $\pm 25\%$	Professional Judgement
Performance evaluation mixture/IND6	12 hours, after analytical run.	Recovery $\pm 25\%$ Combined endrin and 4,4'-DDT degradation $\leq 30\%$ . Endrin or 4,4'-DDT degradation $\leq 20\%$ .	If $\%D > 25$ , then detected results---J, Nondetected results will be estimated UJ if $\%D > 50$ . Use Region III Modifications to NFGs to qualify results if %degradation did not meet..
Holding Time	NA (not applicable)	Water/Soil: 7 days to extraction and 40 days after extraction to analysis date.	Detected Results---J, Nondetected----UJ. If holding time is grossly exceeded: Detected Results---J, Nondetected ---UJ or R (Use professional judgement to determine the reliability of the data and the effect of additional storage on the sample results.
Resolution Check Mixture (RCM) Performance Evaluation Mixture (PEM)	12 hours	RCM: %Resolution $\leq 60\%$ . PEM: %Resolution $\leq 90\%$ .	RCM and/or PEM: Detected Results ---J, Nondetected---Professional Judgement.
Instrument Blank	12 hours, after analytical run.	No target analytes.	Use Region III Modifications to NFGs to qualify the detected results.
Method Blank	Per each extraction batch	No target analytes.	Follow region III Modifications to NFGs to qualify the detected results----B.

**TABLE 3**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Pesticide/PCB QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance QC	Validation Qualifier																					
Surrogate Spikes	Every sample, calibration, blank	<div><div><u>Surrogates</u> decachlorobiphenyl tetrachloro-m-xylene</div><div><u>Water/Soil</u> 60-150% 60-150%</div></div>	Follow Region III Modifications to NFGs.																					
RT windows for target compounds	_____	<div>_____</div> <div>RT of all target compounds in calibration check, samples, blanks, MS, and MSD should be within RT window of mean initial calibration for each column. RT of TCX <math>\pm 0.05</math> of I.C. and RT of DCB <math>\pm 0.10</math> of I.C. of the same column..</div>	Use Professional Judgement to qualify reported compounds "N", or "R". (Follow Region III Modifications to NFGs).																					
Florisil and GPC Cleanup	Under AEC's approach, the laboratory determines whether extract cleanup is necessary.	<div>_____</div> <div><u>Florisil Cleanup up:</u> %R is 80-120 <u>GPC Cleanup:</u> %R is 80-110</div>	Follow Region III Modifications to NFGs for qualifying the data.																					
Matrix Spike and Duplicates	1 per lot/ matrix	<table><thead><tr><th><u>Compound</u></th><th><u>Soil</u> %R    RPD</th><th><u>Water</u> %R    RPD</th></tr></thead><tbody><tr><td>Lindane</td><td>46-127 <math>\leq 50</math></td><td>56-123 <math>\leq 15</math></td></tr><tr><td>Heptachlor</td><td>35-130 <math>\leq 31</math></td><td>40-131 <math>\leq 20</math></td></tr><tr><td>Aldrin</td><td>34-132 <math>\leq 43</math></td><td>40-120 <math>\leq 22</math></td></tr><tr><td>Dieldrin</td><td>31-134 <math>\leq 38</math></td><td>52-126 <math>\leq 18</math></td></tr><tr><td>Endrin</td><td>42-130 <math>\leq 45</math></td><td>56-121 <math>\leq 21</math></td></tr><tr><td>4,4-DDT</td><td>23-134 <math>\leq 50</math></td><td>38-127 <math>\leq 27</math></td></tr></tbody></table>	<u>Compound</u>	<u>Soil</u> %R    RPD	<u>Water</u> %R    RPD	Lindane	46-127 $\leq 50$	56-123 $\leq 15$	Heptachlor	35-130 $\leq 31$	40-131 $\leq 20$	Aldrin	34-132 $\leq 43$	40-120 $\leq 22$	Dieldrin	31-134 $\leq 38$	52-126 $\leq 18$	Endrin	42-130 $\leq 45$	56-121 $\leq 21$	4,4-DDT	23-134 $\leq 50$	38-127 $\leq 27$	Use professional judgement.
<u>Compound</u>	<u>Soil</u> %R    RPD	<u>Water</u> %R    RPD																						
Lindane	46-127 $\leq 50$	56-123 $\leq 15$																						
Heptachlor	35-130 $\leq 31$	40-131 $\leq 20$																						
Aldrin	34-132 $\leq 43$	40-120 $\leq 22$																						
Dieldrin	31-134 $\leq 38$	52-126 $\leq 18$																						
Endrin	42-130 $\leq 45$	56-121 $\leq 21$																						
4,4-DDT	23-134 $\leq 50$	38-127 $\leq 27$																						



**TABLE 4**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Polynuclear Aromatic Hydrocarbons (PAH) QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	Validation Qualifier
Initial Calibration curve	Once every seven analytical run or when the results indicates a problem.	$r \geq 0.995$  $r$ : linear correlation coefficient	Detected Results --- J, Professional Judgement on Nondetected results.
Holding Time	NA	Water/Soil: 7 days to extraction and 40 days after extraction to analysis date.	Detected Results---J or L, Nondetected---UJ or UL (L and UL qualifiers will be used if loss of PAH compounds is evident due to exceeding the holding time criteria). If holding time is grossly exceeded: Detected---J, Nondetected---R or UJ
Daily Calibration standard/ Continuing Calibration standard (CCS)	Every 12 hours after method blank	Response $\pm 25\%$	Detected results --- J, Professional Judgement on Nondetected results.
RT windows for target compounds	-----	RT of all target compounds in CCS, samples, blanks, MS, and MSD should be within RT window obtained from initial calibration.	Use Professional Judgement

**TABLE 4**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Polynuclear Aromatic Hydrocarbons (PAH) QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	Validation Qualifier
Instrument Blank	One at the beginning of the initial calibration, and every 12 hours after each CCS	No target analytes	Professional Judgement
Method Blank	one method per lot	No target analytes	Use Region III Modifications to NFGs for Organic Data Review to qualify the detected results -- -B.
Surrogate	Every sample	<div> <div>Surrogate</div> <div>triphenylene</div> </div> <div> <div>%R - Water *</div> <div>60-124%</div> </div> <div> <div>%R - Soil *</div> <div>30-124%</div> </div>	Detected Results-----J, Nondetected Results-----UJ. If %R<10, then Detected Results ---L, and Nondetected Results-----R
Matrix Spike and Duplicates	1 per lot/ matrix	<div> <div>Compound</div> <div>           acenaphthene            acenaphthylene            anthracene            benzo(a)pyrene            benzo(k)fluorant            hene            fluorene            naphthalene            phenanthrene         </div> </div> <div> <div>Water</div> <div> <div>%Rec.</div> <div>           49-109 /            53-103 /            44-124 /            45-121 /            41-123 /         </div> </div> <div> <div>RPD</div> <div>           ≤30            ≤25            ≤40            ≤38            ≤41         </div> </div> </div> <div> <div>Soil</div> <div> <div>%Rec</div> <div>           0-124            0-139            0-126            27-151            22-132         </div> </div> <div> <div>RPD</div> <div>           ≤50            ≤50            ≤50            ≤62            ≤55         </div> </div> </div> <div> <div>           25-123            0-122            0-155         </div> <div>           ≤49            ≤50            ≤50         </div> </div>	Use professional judgement to determine the need for some qualification of the data using the MS/MSD results in conjunction with other QC criteria.

\* Based on ESE historical recovery of spiked method blanks.

**TABLE 5**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Polychlorinated Terphenyls (PCT) QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance QC	Validation Qualifier
Initial calibration curve single component, multi-component	Set-up, major maintenance	$r \geq 0.995$	Detected results ---J, Professional Judgement on Nondetected results.
Daily calibration standard	12 hours	$r \geq 0.995$	Detected results ---J, Professional Judgement on Nondetected results.
Independent reference standard (calibration check)	Weekly	Recovery $\pm 25\%$	Detected results ---J, Professional Judgement on Nondetected results.
Continuing calibration	12 hours	Response $\pm 25\%$	Detected results ---J, Professional Judgement on Nondetected results.
Holding Time	NA	Water/Soil: 7 days to extraction and 40 days after extraction to analysis date.	Detected results ---J, Non-detected ---UJ. If holding time is grossly exceeded: Detected results ---J, Nondetected ---UJ or R (Use professional judgement to determine the reliability of the data and the effect of additional storage on the sample results.
Instrument blank	12 hours, after analytical run	No target analytes	Use Region III Modifications to NFGs to qualify the detected results.
Method blanks	12 hours	No target analytes	Use Region III Modifications to NFGs to qualify the detected results.

**TABLE 5**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Polychlorinated Terphenyls (PCT) QC Requirements)**

QC Requirements	Frequency of QC Procedure	Acceptance QC				Validation Qualifier
Matrix spike and duplicate	1 per lot	<u>Standard</u>	<u>Water</u> %R <u>RPD</u> 50-150    ≤30 50-150    ≤30	<u>Soil</u> %R <u>RPD</u> 50-150    ≤30 50-150    ≤30	Use Professional Judgement.	
Surrogate spikes	Every sample	<u>Surrogate</u> decachlorobiphenyl tetrachloro-m-xylene	<u>Water</u> 75-125% 75-125%	<u>Soil</u> 75-125% 75-125%		
RT windows for target compounds (Peak Identification)	_____	RT of all target compounds in samples, blanks, and QC samples should be within RT window of mean initial calibration for each column.				Use Professional Judgement.
Acid clean-up/ Silica clean-up	Once per lot. Acid clean-up is required for both water and soil samples.. Silica clean-up however, is only required for soil samples.	QC limits: 80-120% *				Use Professional Judgement.

\* Based on QC limits of other clean-up procedures used in pesticide/PCBs.

**TABLE 6**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Inorganic QC Requirements (TAL Metals))**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	Validation Qualifier
Holding Time	NA	Water & Soil: Metals: 6 months and preserved to pH <2 Mercury: 28 days and preserved to pH <2.	Results > IDL ---- L, Results < IDL ---- UL. If holding times are exceeded by two times the criteria for Hg, Results < IDL --UL. If holding times are grossly exceed for ICP, use professional judgement (Results < IDL ---R)
Initial calibration curve	Major maintenance, instrument modification	$r \geq 0.995$ for all elements	Professional judgement
Daily calibration standard (calibration blank & calibration verification)	12 hours	slope within 10% of initial calibration. %R = 80-120 (for Hg) %R = 90-110 (for metals)	Follow Region III Modifications to NFGs.
Interference Check (ICP only)	Beginning and end of each sample analytical run	Recovery $\pm 20\%$ of the true value.	Follow Region III Modifications to NFGs.
Continuing calibration verification (CCV)	Every 10 samples, end of analytical run	Recovery $\pm 10\%$ of true value	Follow Region III Modifications to NFGs.
Continuing calibration blank (CCB)	Every 10 samples, end of analytical run	No target analytes.	Follow Region III Modifications to NFGs under "BLANK" section.
Serial Dilution (ICP only)	1 per 20 samples	Difference between diluted and undiluted sample $\leq 10\%$ .	Associated data ----J, If negative interference is found, use professional judgement to qualify the data.
Preparation Blank	1 per lot / matrix/ method	No target analytes.	Follow Region III Modifications to NFGs.
Laboratory control sample	1 per lot	Recovery within the established limit.	Use Region III Modifications to NFGs and Professional Judgement.

**TABLE 6**  
**QC Requirements, Criteria, and Validation Qualifiers**  
**(Inorganic QC Requirements (TAL Metals))**

QC Requirements	Frequency of QC Procedure	Acceptance Criteria	Validation Qualifier
Duplicate sample analysis	1 per lot. Field blanks cannot be used for duplicate sample analysis.	RPD $\pm 20\%$ (water) and RPD $\pm 35$ (soil) for sample values $> 5 \times \text{CRDL}$ , RPD $\pm \text{CRDL}$ (water) and RPD $\pm 2 \times \text{CRDL}$ (soil) for sample values $< 5 \times \text{CRDL}$ , including the case when only one of the duplicate sample values is $< 5 \times \text{CRDL}$ .	Results $> \text{IDL}$ -----J, results $< \text{IDL}$ ----UJ.
Matrix Spike and duplicate (Post-Digestion Spikes)	1 per lot. Field blanks cannot be used for duplicate sample analysis.	%R must be within the limits of 75 to 125 percent. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of four or more. The post-digestion /analytical spike recovery for the GFAA metals must be within 85-115%. If the matrix spike recovery limits are not met and the sample concentration does not exceed 4X the spike added, a post-digestion spike must be performed for ICP and flame AA. This criteria is not required for Ag and Hg.	Follow Region III Modifications to NFGs to qualify the data.

\* *Method 7041* (Analysis of soil samples for SB by graphite furnace atomic absorption spectrometry): a minimum of five standards which include a blank, a standard at the lower reporting limit and a standard at the upper reporting limit.  
*Methods 7470 & 7471* (Determination of Hg in liquid waste & soil using CVAA): seven standards which include a blank.  
*Method 6010* (Determination of metals in soil by ICP): a minimum of five standards which consist of a blank, standard at the reporting limit, two intermediate standards [the daily calibration level and a 1:1 dilution of the daily calibration level] and a standard at the upper linear range.  
*Method 6010* (Determination of metals in water by ICP): a calibration blank and five standards.  
*Method 6020* (Determination of metals in water by ICPMS) : a minimum of two standards which include a blank standard.

# **APPENDIX D**

## **Standard Operating Procedure**

The general QA/QC procedure for data validation:

- 1) Data is received by Horne Engineering receptionist, is not opened, and is hand delivered to the Senior Chemist.
- 2) The Senior Chemist logs receipt of data deliverable into a Data Validation Log Book (Attachment I). This book will be maintained in a locked file cabinet in the Senior Chemist's office.
- 3) The Senior Chemist distributes data packages to appropriate chemists. Distribution is based on schedule and content (organic/inorganic). The Senior Chemist will assign a schedule to the chemists at the time the data package is handed off.
- 4) The Senior Chemist notifies the Project Manager of data deliverable and assignments.
- 5) The Review Chemist will perform manual data validation either on summary data or the whole data package depending on Senior Chemist's assignment.
- 6) Based on the evaluation of QC elements, a qualifier will be assigned to the data as appropriate (Table 1- Table 6). The Review Chemist is responsible for completing review and preparing a report within the schedule specified by the Senior Chemist. The Review Chemist will notify the Senior Chemist of all variations in schedule. These variations will be assessed by the Senior Chemist or their impact on the schedule.
- 7) The Review Chemist will hand deliver the Data Validation Report to the Senior Chemist.
- 8) The Senior Chemist will verify the Data Validation Report for clarity, consistency, and accuracy. All technical assessments (especially those involving qualifiers) will be reviewed. Any necessary corrections will be made by the Review Chemist and checked by the Senior Chemist.
- 9) When Senior Chemist concurs with the Review Chemist on the data, they will both sign the completed Data Validation Report.
- 10) The signed report will be presented to the QA officer for the final QC and will be presented to the Project and Program Manager. The managers will release the report by completing a Project Management Approval and Release Form (Attachment II).
- 11) Horne Engineering will log out the report and send copies to the AEC Project Manager.



# **Appendix E**

## **Example of Quality Assurance Report**

## QUALITY ASSURANCE REPORT PREPARATION

### Purpose

The purpose of this procedure is to establish a report format for organic and inorganic data review report writing according to EPA Region III protocol (where applicable).

### Discussion

After completion of data review, the data reviewer will be responsible for compiling review notes (notes obtained from validating each lot of the same site) and writing a report. The outline below describes the steps to follow in preparing the data review report.

### Procedure

#### 1.1 Data Validation Narrative

The validation narrative is for the data user.

- 1.1.1 The first page of the report should be printed on letterhead. The address of the report should include the following information:

Date:           Month DD, Year

Subject:       Organic or Inorganic Data Validation for Each Site (write site name)

From:	Reviewer Name	Oversight Reviewer Name
	Reviewer Title	Reviewer Title

To:            Client Name

#### 1.1.2 Overview

The first section of the report is the overview, and is presented in paragraph form after the title "OVERVIEW". Information in this paragraph should include:

- ▶ Lot Numbers
- ▶ Analytes
- ▶ Number of Samples per Site
- ▶ Matrix
- ▶ The Method under which the laboratory performed the analysis
- ▶ Laboratory Name

### 1.1.3 Summary

The summary section, written below the title "SUMMARY", is a general statement noting whether the samples were successfully analyzed or if there were any analyses determined unsuccessful (e.g., data were qualified unusable).

### 1.1.4 Major Problems

After the section title, "MAJOR PROBLEMS", any problems identified during the validation that seriously affect data usability and any data that are qualified unusable, "R", is noted in this portion of the narrative. Identification of the "Major problems" (including support documentation) will be compiled for each data package of the same site and will be referred as an attachment.

#### Minor Problems

The section title, "MINOR PROBLEMS", is followed by a series of bulleted paragraphs describing biases identified during the data review which may qualify data as "J", "UJ", "K", "L", or "UL". Identification of the "Minor Problems" (including support documentation) will be compiled for each data package of the same site and will be referred as an attachment.

### 1.1.7 Attachments

Under this section, the following lists and their contents is included.

- 1.1.7.1 Glossary of Data Qualifiers -- a listing of all organic/ inorganic data qualifiers used in Region III and their definitions.
- 1.1.7.2 IRDMIS Data Entry (per package/per lot)
- 1.1.7.3 Support Documentation - include all support documentation needed to substantiate the findings described in the narrative as well as "Record of Communication"

# **GLOSSARY OF DATA QUALIFIERS**

## GLOSSARY OF DATA QUALIFIER CODES

### CODES RELATED TO IDENTIFICATION (Confidence concerning presence or absence of compounds)

- U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.
- B = Not detected substantially above the level reported in laboratory or field blanks.
- R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.
- N = Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts. (*Organic only*)
- (NO CODE) = Confirmed identification.

### CODES RELATED TO QUANTITATION (can be used for both positive results and sample QUANTITATION limits):

- J = Analyte present. Reported value may not be accurate or precise.
- K = Analyte present. Reported value may be biased high. Actual value is expected lower.
- L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.
- UJ = Not detected, quantitation limit may be inaccurate or imprecise.
- UL = Not detected, quantitation limit is probably higher.

### OTHER CODES

- Q = No analytical result.
- NJ = Qualitative identification questionable due to poor resolution. Presumptively present at approximate quantity (*Organic only*)
- \* = Results reported from diluted analysis. (*Inorganic Only*)

## DATA SUMMARY TABLES

TABLE 1, Page 1 of 1  
 SITE NAME, LOT NUMBER  
 REVIEW DATE  
 PESTICIDE/PCB ANALYSIS  
 WATER ANALYTICAL RESULTS (ug/L)

Sample Location						
Date Sampled						
Dilution Factor						
Remarks						
PESTICIDE/PCB COMPOUND	CRQL					
Aldrin						
alpha-BHC						
beta-BHC						
delta-BHC						
gamma-BHC (Lindane)						
Chlordane						
alpha-Chlordane						
gamma-Chlordane						
Dieldrin						
4,4'-DDD						
4,4'-DDE						
4,4'-DDT						
Endosulfan I						
Endosulfan II						
Endosulfan sulfate						
Endrin						
Endrin aldehyde						
Endrin ketone						
Heptachlor						
Heptachlor epoxide						
Methoxychlor						
Toxaphene						
Aroclor-1016						
Aroclor-1221						
Aroclor-1232						
Aroclor-1242						
Aroclor-1248						
Aroclor-1254						
Aroclor-1260						

TABLE 2, Page 1 of 1  
 SITE NAME, LOT NUMBER  
 REVIEW DATE  
 PESTICIDE/PCB ANALYSIS  
 SOIL ANALYTICAL RESULTS (ug/Kg)

Sample Location					
Date Sampled					
Percent Solids					
Dilution Factor					
Remarks					
PESTICIDE/PCB COMPOUND					
Aldrin alpha-BHC beta-BHC delta-BHC gamma-BHC (Lindane) Chlordane alpha-chlordane gamma-chlordane Dieldrin 4,4'-DDD 4,4'-DDE 4,4-DDT Endosulfan I Endosulfan II Endosulfan sulfate Endrin Endrin aldehyde Endrin ketone Heptachlor Heptachlor epoxide Methoxychlor Toxaphene Aroclor-1016 Aroclor-1221 Aroclor-1232 Aroclor-1242 Aroclor-1248 Aroclor-1254 Aroclor-1260					



**TABLE 1, Page 1 of 1**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**VOLATILE ANALYSIS**  
**WATER ANALYTICAL RESULTS (ug/L)**

<b>Sample Location</b>						
<b>Date Sampled</b>						
<b>Dilution Factor</b>						
<b>Remarks</b>						
<b>VOLATILE (VOA) COMPOUND</b>	<b>CRQL</b>					
Acetone						
Benzene						
Bromodichloromethane						
Bromoform						
Bromomethane						
2-Butanone						
Carbon disulfide						
Carbon tetrachloride						
Chlorobenzene						
Chloroethane						
2-Chloroethyl vinyl ether						
Chloroform						
Chloromethane						
Dibromochloromethane						
1,1-Dichloroethane						
1,2-Dichloroethane						
1,1-Dichloroethene						
1,2-Dichloroethene (total)						
1,2-Dichloropropane						
cis-1,3-Dichloropropene						
trans-1,3-Dichloropropene						
Ethylbenzene						
2-Hexanone						
4-Methyl-2-pentanone						
Methylene chloride						
Styrene						
1,1,2,2-Tetrachloroethane						
Tetrachloroethene						
Toluene						
1,1,1-Trichloroethane						
1,1,2-Trichloroethane						
Trichloroethane						
Vinyl Acetate						
Vinyl chloride						
Xylenes (total)						

TABLE 2, Page 1 of 1  
 SITE NAME, LOT NUMBER  
 REVIEW DATE  
 VOLATILE ANALYSIS  
 SOIL ANALYTICAL RESULTS (ug/Kg)

Sample Location					
Date Sampled					
Percent Moisture					
Dilution Factor					
Remarks					
<b>VOLATILE (VOA) COMPOUND</b>					
Acetone					
Benzene					
Bromodichloromethane					
Bromoform					
Bromomethane					
2-Butanone					
Carbon disulfide					
Carbon tetrachloride					
Chlorobenzene					
Chloroethane					
2-Chloroethyl vinyl ether					
Chloroform					
Chloromethane					
Dibromochloromethane					
1,1-Dichloroethane					
1,2-Dichloroethane					
1,1-Dichloroethene					
1,2-Dichloroethene (total)					
1,2-Dichloropropane					
cis-1,3-Dichloropropene					
trans-1,3-Dichloropropene					
Ethylbenzene					
2-Hexanone					
4-Methyl-2-pentanone					
Methylene chloride					
Styrene					
1,1,2,2-Tetrachloroethane					
Tetrachloroethene					
Toluene					
1,1,1-Trichloroethane					
1,1,2-Trichloroethane					
Trichloroethane					
Vinyl acetate					
Vinyl chloride					
Xylenes (total)					

**TABLE 1, Page 1 of 2**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**SEMIVOLATILE ANALYSIS**  
**WATER ANALYTICAL RESULTS (ug/L)**

<b>Sample Location</b>						
<b>Date Sampled</b>						
<b>Dilution Factor</b>						
<b>Remarks</b>						
<b>SEMIVOLATILE (SVOA) COMPOUND</b>	<b>CRQL</b>					
Phenol bis(2-chloroethyl)ether 2-Chlorophenol 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene Benzyl alcohol 2-Methylphenol 2,2'-oxybis(1-chloropropane) 4-Methylphenol N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene Isophorone 2-Nitrophenol 2,4-Dimethylphenol bis(2-Chloroethoxy)methane 2,4-Dinitrophenol 2,4-Dichlorophenol Benzoic acid 1,2,4-Trichlorobenzene Naphthalene 4-Chloroaniline Hexachlororbutadiene 4-Chloro-3-methylphenol 2-Methylnaphthalene Hexachlorocyclopentadiene 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 2-Chloronaphthalene 2-Nitroaniline 3-Nitroaniline Dimethylphthalate Acenaphthylene Acenaphthene						

**TABLE 1, Page 2 of 2**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**SEMIVOLATILE ANALYSIS**  
**WATER ANALYTICAL RESULTS (ug/L)**

<b>Sample Location</b>						
<b>Date Sampled</b>						
<b>Dilution Factor</b>						
<b>Remarks</b>						
<b>SEMIVOLATILE (SVOA) COMPOUND</b>	<b>CRQL</b>					
2,6-Dinitrotoluene						
4-Nitrophenol						
Dibenzofuran						
2,4-Dinitrotoluene						
Diethylphthalate						
4-Chlorophenyl-phenylether						
Fluorene						
4-Nitroaniline						
4,6-Dinitro-2-methylphenol						
N-Nitrosodiphenylamine(1)						
4-Bromophenyl-phenylether						
Hexachlorobenzene						
Pentachlorophenol						
Phenanthrene						
Anthracene						
Carbazole						
Di-n-butylphthalate						
Fluoranthene						
Pyrene						
Butylbenzylphthalate						
3,3'-Dichlorobenzidine						
Benzo(a)anthracene						
Chrysene						
bis(2-Ethylhexyl)phthalate						
Di-n-octylphthalate						
Benzo(a)pyrene						
Benzo(b)fluoranthene						
Benzo(k)fluoranthene						
Indeno(1,2,3-cd)pyrene						
Dibenz(a,h)anthracene						
Benzo(g,h,i)perylene						

TABLE 2, Page 1 of 2  
 SITE NAME, LOT NUMBER  
 REVIEW DATE  
 SEMIVOLATILE ANALYSIS  
 SOIL ANALYTICAL RESULTS (ug/Kg)

Sample Location					
Date Sampled					
Percent Moisture					
Dilution Factor					
Remarks					
SEMEVOLATILE (SVOA) COMPOUND					
Phenol bis(2-chloroethyl)ether 2-Chlorophenol 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene Benzyl alcohol 2-Methylphenol 2,2'-oxybis(1-chloropropane) 4-Methylphenol N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene Isophorone 2-Nitrophenol 2,4-Dimethylphenol bis(2-Chloroethoxy)methane 2,4-Dinitrophenol 2,4-Dichlorophenol Benzoic acid 1,2,4-Trichlorobenzene Naphthalene 4-Chloroaniline Hexachlororbutadiene 4-Chloro-3-methylphenol 2-Methylnaphthalene Hexachlorocyclopentadiene 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 2-Chloronaphthalene 2-Nitroaniline 3-Nitroaniline Dimethylphthalate Acenaphthylene Acenaphthene					

TABLE 2, Page 2 of 2  
 SITE NAME, LOT NUMBER  
 REVIEW DATE  
 SEMIVOLATILE ANALYSIS  
 SOIL ANALYTICAL RESULTS (ug/Kg)

Sample Location					
Date Sampled					
Percent Moisture					
Dilution Factor					
Remarks					
SEMEVOLATILE (SVOA) COMPOUND					
2,6-Dinitrotoluene					
4-Nitrophenol					
Dibenzofuran					
2,4-Dinitrotoluene					
Diethylphthalate					
4-Chlorophenyl-phenylether					
Fluorene					
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine(1)					
4-Bromophenyl-phenylether					
Hexachlorobenzene					
Pentachlorophenol					
Phenanthrene					
Anthracene					
Carbazole					
Di-n-butylphthalate					
Fluoranthene					
Pyrene					
Butylbenzylphthalate					
3,3'-Dichlorobenzidine					
Benzo(a)anthracene					
Chrysene					
bis(2-Ethylhexyl)phthalate					
Di-n-octylphthalate					
Benzo(a)pyrene					
Benzo(b)fluoranthene					
Benzo(k)fluoranthene					
Indeno(1,2,3-cd)pyrene					
Dibenz(a,h)anthracene					
Benzo(g,h,i)perylene					

**TABLE 1, Page 1 of 1**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**INORGANIC ANALYSIS**  
**WATER ANALYTICAL RESULTS (ug/L)**

<b>Sample Location</b>							
<b>Date Sampled</b>							
<b>Dilution Factor</b>							
<b>Remarks</b>							
<b>INORGANIC ELEMENTS</b>	<b>Instrument</b>	<b>Detection Limits (Ug/L)</b>					
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

**TABLE 2, Page 1 of 1**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**INORGANIC ANALYSIS**  
**SOIL ANALYTICAL RESULTS (mg/Kg)**

<b>Sample Location</b>						
<b>Date Sampled</b>						
<b>% Solid</b>						
<b>Dilution Factor</b>						
<b>Remarks</b>						
<b>INORGANIC ELEMENTS</b>	<b>Method</b>					
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						



**TABLE 1, Page 1 of 1**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**POLYNUCLEAR AROMATIC HYDROCARBON**  
**WATER ANALYTICAL RESULTS (ug/L)**

<b>Sample Location</b>						
<b>Date Sampled</b>						
<b>Dilution Factor</b>						
<b>Remarks</b>						
<b>POLYNUCLEAR AROMATIC HYDROCARBON</b>	<b>CRQL</b>					
Acenaphthene						
Acenaphthylene						
Anthracene						
Benzo(a)anthracene						
Benzo(a)pyrene						
Benzo(b)fluoranthene						
Benzo(g,h,i)perylene						
Benzo(k)fluoranthene						
Chrysene						
Dibenzo(a,h)anthracene						
Fluoranthene						
Fluorene						
Indeno(1,2,3-cd)pyrene						
Naphthalene						
1-Methylnaphthalene						
2-Methylnaphthalene						
Phenanthrene						
Pyrene						

TABLE 2, Page 1 of 1  
 SITE NAME, LOT NUMBER  
 REVIEW DATE  
 POLYNUCLEAR AROMATIC HYDROCARBON  
 SOIL ANALYTICAL RESULTS (ug/Kg)

Sample Location					
Date Sampled					
Percent Solids					
Dilution Factor					
Remarks					
POLYNUCLEAR AROMATIC HYDROCARBON					
Acenaphthene Acenaphthylene Anthracene Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(g,h,i)perylene Benzo(k)fluoranthene Chrysene Dibenzo(a,h)anthracene Fluoranthene Fluorene Indeno(1,2,3-cd)pyrene Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene Phenanthrene Pyrene					

**TABLE 1, Page 1 of 1**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**POLYCHLORINATED TERPHENYL ANALYSIS**  
**WATER ANALYTICAL RESULTS (UG/L)**

<b>Sample Location</b>						
<b>Date Sampled</b>						
<b>Dilution Factor</b>						
<b>Remarks</b>						
<b>POLYCHLORINATED TERPHENYLS</b>	<b>CRQL</b>					
AROCLOR-5432 AROCLOR- 5442 AROCLOR-5460						

**TABLE 2, Page 1 of 1**  
**SITE NAME, LOT NUMBER**  
**REVIEW DATE**  
**POLYCHLORINATED TERPHENYL ANALYSIS**  
**SOIL ANALYTICAL RESULTS (ug/kg)**

<b>Sample Location</b>					
<b>Date Sampled</b>					
<b>% Moisture</b>					
<b>Dilution Factor</b>					
<b>Remarks</b>					
<b>POLYCHLORINATED TERPHENYLS</b>					
AROCLOR-5432 AROCLOR-5442 AROCLOR-5460					

## **SUPPORT DOCUMENTATION**

# RECORD OF COMMUNICATION

NAME : -----

CONTACT REC'D. VIA: Phone : -----Fax : -----Mail : -----  
Memo : -----Other : -----

DATE/TIME OF CONTACT : -----

INITIATED BY : Lab : -----Region : -----  
AEC : -----Other : -----

CONTACT NAME / ORG. / PHONE # : -----

Lab -----	Contract # -----	Lot # -----
Method -----	Validation Level -----	Affected Samples:-----

DISCUSSION / ISSUE : -----  
-----  
-----  
-----

RESOLUTION : -----  
-----  
-----  
-----

Completed Date/Time : -----

Distribution :

# **Attachment I**

## **Data Validation Log Sheet**

## Data Validation Log Sheet

Electronic Data Date Received: -----

Project No.: -----

Hardcopy Data Date Received: -----

Client: -----

Special Instructions: -----  
-----  
-----  
-----

### Due Dates:

Review Chemist : -----

Project Manager: -----

Project Chemist : -----

Client: -----

Lot / SDG #	Matrix	No. of Samples	Fraction/Method	Review Hours	QC Hours



# **Attachment II**

## **Project Management Review & Approval Form Release**

## Project Management Review & Approval Form Release

Date: -----

Project Name: -----

Project Manager: -----

Program Manger: -----

QC Criteria	Comments/Deviations
<input type="checkbox"/> Data Validation Report contains all appropriate sections in the correct order.	
<input type="checkbox"/> Communication Records and Resubmissions are included in an appendix.	
<input type="checkbox"/> Project Chemist has QC'd the Data Validation Report.	
<input type="checkbox"/> Copies of electronic deliverables are included (both hard and electronic copies)	

This report meets Horn Engineering Quality Control requirements and is approved for release.

-----  
Horne Engineering Project Manager

-----  
Date

-----  
Horne Engineering Program Manager

-----  
Date

# **Appendix F**

## **Draft Woodbridge Research Facility Data Validation Schedule**

# Final Woodbridge Research Facility Data Validation Project Schedule

